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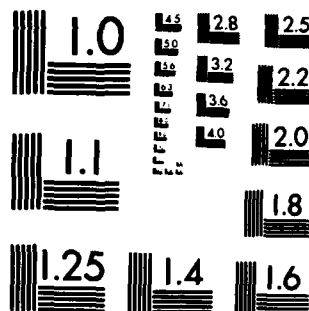
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1,3-DIPOLAR CYCLOADDITION REACTIONS OF LOW VALENT  
METAL-CARBONYL COMPLEXES WITH ARYLNITRILE-N-OXIDES

By

John A. Walker, Carolyn B. Knobler and M. Frederick Hawthorne\*

Prepared for Publication

in

Journal of the American Chemical Society

Department of Chemistry and Biochemistry  
University of California  
Los Angeles, California 90024

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ABSTRACT

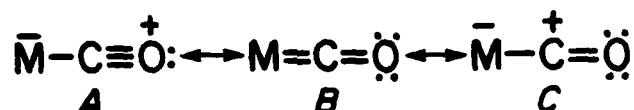
Several low valent metal carbonyl complexes are reported to react with aryl-nitrile-N-oxides to form  $(O-N=(Ar)C-M-C(O))$  from a 1,3-dipolar cycloaddition of aryl-nitrile-N-oxide to a  $M-C(O)$  bond. Complexes  $[Et_4N][(\text{Ph}_3P)(CO)RhC_2B_9H_{11}]$  (1a; 3,1,2 isomer and 1b; 2,1,7 isomer) react with benzonitrile-N-oxide to produce cycloadducts 2a and 2b, respectively. Similarly  $K[18\text{-crown-6}]$  1a and  $[Et_4N]$  1b react with *m*-fluorobenzonitrile-N-oxide to produce 3a and 3b, respectively. Analytical and spectral characterization of the new cycloadducts was supported by the X-ray diffraction study of the  $[PPN]^+$  salt of 3b. Reactions of the cycloadducts with CO are described. The complexes  $K[18\text{-crown-6}]M(CO)_5$  ( $M=Re, Mn$ ) react with benzonitrile-N-oxide to produce cycloadducts 5a and 5b, respectively. Complex 5a is quite stable, but 5b is exceedingly unstable.  $(\eta^5-C_5(CH_3)_5)Rh(CO)_2$  reacts with *p*-chlorobenzonitrile-N-Oxide to produce an intermediate cycloadduct, 8, which can be converted to the known dimeric complex  $[(\eta^5-C_5(CH_3)_5)Rh(\mu-CO)]_2$ .

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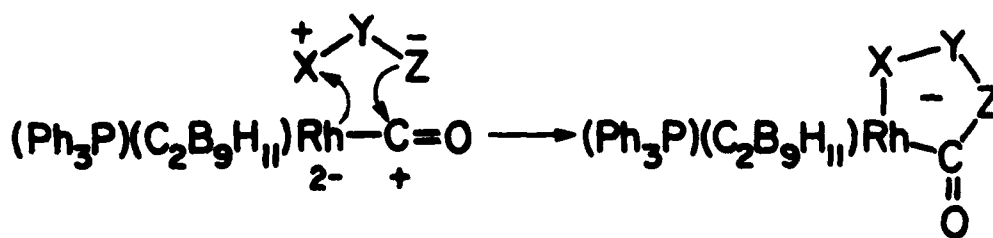


Recently chemists have begun to recognize and exploit isolobal<sup>1a</sup> relationships of C=C and M=C moieties. For example, Stone and coworkers have used this apparent bonding analogy to generate many heteronuclear metal clusters from zero valent platinum reagents or other low valent metal species and Fischer-type carbenes and carbynes.<sup>1b</sup> While these studies have focused on well defined metal-carbon multiple bonds, another class of metal complexes generally believed to contain metal-carbon multiple bond character<sup>2</sup>, the low valent metal carbonyl complexes, has not received similar attention. We report here a new reaction involving this incipient M=C bond.

Bonding in metal carbonyl complexes can be qualitatively described by A, B and C.



Structures A and C, which do not involve back-bonding, suggest the presence of a nucleophilic metal atom and an electrophilic carbon atom which, when employed together, might lead to reactions of certain metal carbonyl complexes with 1,3-dipolar molecules. Optimally, an electron rich metal carbonyl complex capable of increasing its coordination number from five to six would be expected to be particularly reactive. The two isomeric rhodacarborane anions  $[(Ph_3P)(CO)RhC_2B_9H_{11}]^-$  (1a; 3,1,2-isomer and 1b; 2,1,7-isomer), reported in the preceding communication<sup>3</sup>, possess the key electronic and structural features necessary for such a [3 + 2] cycloaddition reaction. viz:

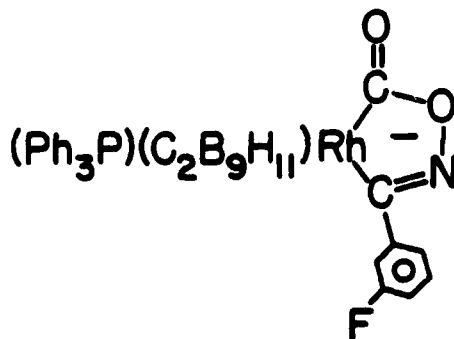




Indeed the classical 1,3-dipolar molecule, benzonitrile-N-oxide<sup>4</sup>, is reactive towards 1a and 1b; we report the complete characterization of the resultant cycloadducts and the reactivity of several other low valent metal carbonyl complexes towards aryl nitrile-N-oxides.

Dichloromethane solutions of tetraethylammonium salts of 1a and 1b smoothly reacted with 1.3 molar equivalents of benzhydroxamic acid chloride (m.p. 45-48°; caution: skin irritant)<sup>5</sup> upon warming from -78° to 0°C in the presence of anhydrous K<sub>2</sub>CO<sub>3</sub> to produce cycloadducts 2a and 2b, respectively. Complex 2b was isolated as a tetraethylammonium salt (75% yield) by filtration and addition of excess diethylether at 0°. An analytical sample was obtained by recrystallization from CH<sub>3</sub>CN/(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O at -20° C. Pure NEt<sub>4</sub><sup>+</sup> salt of complex 2a could not be isolated due to product decomposition. Similarly K[18-crown-6] 1a and NEt<sub>4</sub><sup>+</sup> 1b react with m-fluorobenzhydroxamic acid chloride to generate analytically pure cycloadducts K[18-crown-6] 3a and NEt<sub>4</sub><sup>+</sup> 3b. Cycloadducts 2a, 2b, 3a and 3b all display strong absorptions<sup>6</sup> in the region of 1670-1640 cm<sup>-1</sup> (exocyclic C=O) and medium to weak bands in the 1540-1520 cm<sup>-1</sup> region (C=N).

An X-ray diffraction study was carried out on the [PPN]<sup>+</sup> salt of 3b<sup>7</sup>; a projection of the anion is shown in Figure 1. Bond lengths within the metallacycle are not unusual for the valence bond representation shown below which can be described as a 4-rhodaisoxazolin-5-one.





The cycloadducts release carbon dioxide and benzonitrile or *m*-fluorobenzonitrile upon warming. When acetone solutions of the cycloadducts are maintained at 40° C, the pale-yellow color of the metallacycle is replaced by a deep red. The 81.02 MHz  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of the red solution generated from 3a showed  $\text{K}[18\text{-crown-6}][3,3\text{-(Ph}_3\text{P)}_2\text{-3,1,2-RhC}_2\text{B}_9\text{H}_{11}]$  to be the only phosphorus containing species present. The nature of the additional products has not been determined. Complexes 2b and 3b undergo similar thermal decomposition generating  $[(\text{C}_2\text{H}_5)_4\text{N}][2,2\text{-(Ph}_3\text{P)}_2\text{-2,1,7-RhC}_2\text{B}_9\text{H}_{11}]$ . When the decomposition reaction was performed at 40° C under an atmosphere of CO, both 3a and 3b quantitatively regenerated 1a and 1b, respectively.

The 1,3-dipolar cycloaddition reaction of aryl nitrile-N-oxides could be extended to other low valent metal carbonyl complexes. Slow addition of an ether solution of benzonitrile-N-oxide to a THF solution of  $\text{K}[18\text{-crown-6}][\text{Re}(\text{CO})_5]^6$  at -50° C generates a white cycloadduct, 5a,<sup>6</sup> analogous to 2a. Complex 5a is isolable (60-65%) as a remarkably stable salt, m.p. 155° C (dec). The IR spectrum of 5a indicates that it has the expected metallacyclic structure analogous to 2a or 2b. The reaction of benzhydroxamic acid chloride with  $\text{K}[18\text{-crown-6}][\text{Mn}(\text{CO})_5]^6$  in the presence of anhydrous potassium carbonate at -20° C produces the white cycloadduct, 5b.<sup>6</sup> IR and  $^1\text{H}$  NMR spectroscopy indicate that 5b is exactly analogous to 5a; however, due to the low thermal stability of 5b, satisfactory analytical data were not obtained.

Trimethylamine-N-oxide has been commonly used to remove terminal carbonyl ligands (as  $\text{CO}_2$ ), producing unsaturated and reactive metal centers.<sup>8</sup> For example, the complex  $(\eta^5\text{-C}_5(\text{CH}_3)_5)\text{Rh}(\text{CO})_2$  6, has been converted to the dimeric complex  $[(\eta^5\text{-C}_5(\text{CH}_3)_5)\text{Rh}(\mu\text{-CO})]_2$ , 7, by  $(\text{CH}_3)_3\text{NO}$  in high yield.<sup>9</sup> Complex 6 reacts with *p*-chlorobenzonitrile-N-oxide at -40° C to produce yellow-white cycloadduct, 8. Infrared and  $^1\text{H}$  NMR spectra indicate that 8 has the anticipated metallacycle structure. Complex 8, thermally unstable (dec. 55° C), slowly produces dimeric complex 7 in solution at 25° C, a transformation which underscores the potential of this methodology for removing coordinated carbon monoxide ligands under exceedingly mild conditions.



Although this report apparently marks the first occasion that a nitrile oxide has been incorporated into a transition metal complex, another class of 1,3-dipolar molecules, aryl and aroyl azides, were long ago reacted with Vaska's complex, trans-(Ph<sub>3</sub>P)<sub>2</sub>(CO)IrCl, to produce one of the first dinitrogen complexes, trans-(Ph<sub>3</sub>P)<sub>2</sub>(N<sub>2</sub>)IrCl.<sup>10</sup> In light of our current findings, the mode of formation of this iridium-dinitrogen complex may involve 1,3-dipolar addition of the organic azide to the Ir-C(O) dipole in trans-(Ph<sub>3</sub>P)<sub>2</sub>(CO)IrCl to form a five-coordinate metallacycle (Ph<sub>3</sub>P)<sub>2</sub>(Cl)ArC(O)N=N=N-Ir-C(O) which subsequently decomposes to the observed products. The formation of the dinitrogen complex, trans-(Ph<sub>3</sub>P)<sub>2</sub>(N<sub>2</sub>)IrCl, was originally proposed<sup>10</sup> to arise from a 1,3-oxidative addition of aroyl azide to the Ir center in trans-(Ph<sub>3</sub>P)<sub>2</sub>(N<sub>2</sub>)IrCl followed by reductive elimination of aroyl isocyanate to generate dinitrogen complex.

The structural characterization of cycloadduct 3b demonstrates the first unequivocal 1,3-dipolar addition to a polarized M=C bond. Decomposition of the cycloadducts described here under mild conditions portends a powerful route to unsaturated, low valent metal complexes and clusters. The utility of this chemistry lies in the exploitation of the unsaturated metal complexes formed by this means and probable application of nitrile oxide reactions to other metal-carbon and metal-metal bonded species. Work in this and related areas continues and will be reported at a later date.

#### ACKNOWLEDGEMENT:

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Figure 1.



SUPPLEMENTARY MATERIAL

2a IR (nujol) 1650(s,  $\nu_{C=O}$ ), 1520(m,  $\nu_{C=N}$ )  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CD}_3\text{COCD}_3$ , 200K)

26.8 ppm (d,  $J_{\text{Rh-P}} = 122$  Hz). (Referenced to external 85%  $\text{D}_3\text{PO}_4$ )

2b Anal. ( $\text{C}_{36}\text{H}_{51}\text{N}_2\text{P}_1\text{RhB}_9\text{O}_2$ ) C, H, P, N, Rh, B. IR (nujol) 1680 (s,  $\nu_{C=O}$ ),

(m,  $\nu_{C=N}$ ).  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CD}_2\text{Cl}_2$ , 200K) 26.5 ppm (d,  $J_{\text{Rh-P}} = 120$  Hz).

3a Anal. ( $\text{C}_{40}\text{H}_{54}\text{NPRhB}_9\text{FKO}_8$ ) C, H, N, P, Rh, B, F, K. IR (nujol) 1650

(s,  $\nu_{C=O}$ ), 1520 (m,  $\nu_{C=N}$ ).  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CD}_3\text{COCD}_3$ , 200K) 25.8 ppm

(d,  $J_{\text{Rh-P}} = 118$  Hz).

3b Anal. ( $\text{C}_{36}\text{H}_{50}\text{N}_2\text{P}_1\text{Rh}_1\text{B}_9\text{F O}_2$ ) C, H, N, P, Rh, B, F. IR (nujol) 1650 (s,  $\nu_{C=O}$ ),

1520 (w,  $\nu_{C=N}$ ).  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CD}_3\text{COCD}_3$ , 200K) 24.5 ppm (d,  $J_{\text{Rh-P}} = 120$  Hz).

$\text{K}[18\text{-crown-6}][\text{M}(\text{CO})_5]$  (M = Mn, Re) were produced by reduction of the corresponding

$\text{M}_2(\text{CO})_{10}$  complexes in the THF with K(Hg) in the presence of 18-crown-6.

5a Anal. (calcd for  $\text{C}_{24}\text{H}_{29}\text{N Re MO}_{12}$ ) C, H, K, K; Re calcd, 24.87; found 24.39;

Ir(nujol) 1640  $\text{cm}^{-1}$  ( $\nu_{C=O}$ ) 1475  $\text{cm}^{-1}$  ( $\nu_{C-N}$ )

5b IR(nujol) 1640  $\text{cm}^{-1}$  ( $\nu_{C=O}$ ), 1495  $\text{cm}^{-1}$  ( $\nu_{C=N}$ )



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ABSTRACT

Several low valent metal carbonyl complexes are reported to react with aryl-nitrile-N-oxides to form  $(\text{O}=\text{N}=\text{Ar})\text{C}-\text{M}-\text{C}(\text{O})$  from a 1,3-dipolar cycloaddition of aryl-nitrile-N-oxide to a  $\text{M}-\text{C}(\text{O})$  bond. Complexes  $[\text{Et}_4\text{N}][(\text{Ph}_3\text{P})(\text{CO})\text{RhC}_2\text{B}_9\text{H}_{11}](\underline{1a}; 3,1,2 \text{ isomer and } \underline{1b}; 2,1,7 \text{ isomer})$  react with benzonitrile-N-oxide to produce cycloadducts 2a and 2b, respectively. Similarly  $\text{K}[18\text{-crown-6}] \underline{1a}$  and  $[\text{Et}_4\text{N}] \underline{1b}$  react with *m*-fluorobenzonitrile-N-oxide to produce 3a and 3b, respectively. Analytical and spectral characterization of the new cycloadducts was supported by the X-ray diffraction study of the  $[\text{PPN}]^+$  salt of 3b. Reactions of the cycloadducts with CO are described. The complexes  $\text{K}[18\text{-crown-6}]\text{M}(\text{CO})_5 (\text{M}=\text{Re}, \text{Mn})$  react with benzonitrile-N-oxide to produce cycloadducts 5a and 5b, respectively. Complex 5a is quite stable, but 5b is exceedingly unstable.  $(\eta^5\text{-C}_5(\text{CH}_3)_5)\text{Rh}(\text{CO})_2$  reacts with *p*-chlorobenzonitrile-N-Oxide to produce an intermediate cycloadduct, 8, which can be converted to the known dimeric complex  $[(\eta^5\text{-C}_5(\text{CH}_3)_5)\text{Rh}(\mu\text{-CO})]_2$ .



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